

DOCKET NO: 208608US0PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :
THOMAS DANIEL, ET AL. : EXAMINER: METZMAIER
SERIAL NO: 09/831,915 :
FILED: MAY 25, 2001 : GROUP ART UNIT: 1796
FOR: HYDROGELS CAPABLE OF :
ABSORBING AQUEOUS FLUIDS :

REPLY BRIEF

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

SIR:

Responsive to the Examiner's Answer mailed January 28, 2008, please
consider the following:

The Examiner adheres to the rejection of (A) Claims 1-3, 6, 7, 10-14, 16, 18,
20-21 and 23-24 under 35 U.S.C. § 103(a) over Procter & Gamble (WO 97/46195) in
view of Trinh et al (US 5,429,628) and Modern Superabsorbent Polymer Technology
and (B) Claims 4-5 and 17 under 35 U.S.C. § 103(a) over Procter & Gamble (WO
97/46195) in view of Trinh et al (US 5,429,628).

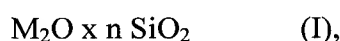
These rejections are again traversed.

Claims 1, 10 and 18 are independent. Claims 1 and 18 relate to a dried
hydrogel and Claim 10 relates to a method of making a hydrogel.

The present invention as set forth in **Claim 1** relates to a dried hydrogel,
prepared by

polymerizing an olefinically unsaturated carboxylic acid or its salts in a
polymerization reaction mixture;

**admixing the polymerization reaction mixture, before, during or after the
polymerization and before drying, with an alkali metal silicate** of the general
formula I



wherein M is an alkali metal and n is from 0.5 to 4;

postcrosslinking a resulting polymer;

thereby obtaining a hydrogel containing said postcrosslinked polymer; and

drying said hydrogel at an elevated temperature, to obtain said dried hydrogel;

wherein said postcrosslinking is effected by a crosslinker which is a

compound containing two or more groups that form covalent bonds with the carboxyl
groups of said polymer.

Procter & Gamble, Trinh et al and Modern Superabsorbent Polymer
Technology fail to disclose or suggest, alone or in combination, a dried hydrogel as
claimed or a process of preparing a dried hydrogel as claimed in which a
polymerization reaction mixture, before, during or after the polymerization and
before drying, is admixed with an alkali metal silicate. In addition, Procter &
Gamble, Trinh et al lack a disclosure of **post-crosslinking**.

The Examiner has stated that “It is reasonable to conclude.....that said admixing process stepsare prior to drying ...”. Examiner’s Answer at page 8, last paragraph. Applicants disagree.

Procter & Gamble discloses an odour control system with silica, AGM and zeolites (page 6, 2nd paragraph). The term “AGM” means absorbent gelling material and is used as synonym for dried hydrogels or superabsorbent polymers. However, **AGM is not a polymerization mixture before drying**. In fact, the Examiner stated at page 3 of the Office Action of July 16, 2007, that AGM is a dried hydrogel. **Since AGM is already a ready made hydrogel which is dried it is impossible to perform a step in Procter & Gamble in which a polymerization mixture is mixed with an alkali metal silicate before drying. Therefore such step is not suggested or motivated.**

Further, Procter & Gamble discloses that in one embodiment, the odour control system is produced using spray drying, spray mixing or agglomeration (page 6, last paragraph). **These techniques relate to the processing of AGM** with binder materials such as silica. However, there is **no disclosure or suggestion that a polymerization mixture before drying be used and admixed with the alkali metal silicate.**

Similarly, Example 9 of *Trinh et al* relates to mixing of AGM with zeolite. Example 9 and col. 16, line 47 et seq. do not provide a disclosure or suggestion that a polymerization mixture before drying be used and admixed with the alkali metal silicate.

Moreover, in Procter & Gamble the silica acts as a binder (page 6, last paragraph) for the AGM particles. However, the silica is **NOT INSIDE THE AGM** particles but **is outside the particles to make them bind**. This is consistent with the

fact that, as stated above, AGM is a ready made dried hydrogel that is mixed with the binder but not in such a way that the binder goes inside the AGM particles.

According to Procter & Gamble, the silica can be distributed homogeneously throughout the absorbent article (page 7, fourth paragraph). **This refers to the entire absorbent article not to a distribution within AGM particles.** Absorbent articles are i.e. sanitary napkins (page 1, first paragraph of Procter & Gamble) and **are not the superabsorbent particles themselves**. Just because the articles have a distribution of silicate on macroscale, does not mean that the single AGM particles contained in the article have silicate distributed therein. The silicate in Procter & Gamble is a binder and thus sits outside the AGM particles to bind them. If the silicate was inside the AGM particles how would it bind them?

In this context, the Examiner's statement at page 12, 1st full paragraph of the Examiner's Answer is misplaced. Applicants argument regarding sanitary napkins refers to the disclosure of Procter & Gamble.

Further, Applicants wish to draw the Examiner's attention to page 6, 1st paragraph, of Procter & Gamble, which states that the odour control system may comprise in addition to silica, additional odour control agents such as zeolites. In other words, mixtures of silica and zeolite are what Procter & Gamble discloses. In paragraph 3 at page 6 of Procter & Gamble, AGM is used together with silica and zeolite.

However, paragraph 3 at page 6 of Procter & Gamble refers to a different embodiment. The particulates, granulates, flakes, noodles, and exudates (page 6, third paragraph) refers to mixtures of silica and zeolite only. There is no AGM in this mixture. Notably, this paragraph refers to a different embodiment than paragraph 2 at

page 6 of Procter & Gamble. This disclosure does not mean particles comprising superabsorbent polymers having silica inside.

In the present invention, as a result of mixing the polymerization mixture with the alkali metal silicate **before drying**, the alkali metal silicate is necessarily distributed inside the particles of the gel that are obtained. This is evidenced by the Declaration of Dr. Manfred Essig which was filed at the USPTO on May 9, 2005. Thus, the dried hydrogel particles of the present invention are different from the AGM particles or the sanitary napkins of Procter & Gamble. While it is clear from Procter & Gamble that the AGM does not include silica inside, the Declaration shows that the dried hydrogel has silica distributed inside. Thus, contrary to the Examiner's position at page 9, 2nd and 3rd full paragraph of the Examiner's Answer, it has been established that the dried hydrogel of the present invention is **patentably distinct** from the odour control system of Procter & Gamble.

In addition, Procter & Gamble, Trinh et al lack a disclosure of **post-crosslinking**. While Modern Superabsorbent Polymer Technology generally discloses the use of post-cross-linking, there is no disclosure that a compound such as an alkali metal silicate is first mixed with a polymer to be post-crosslinked. However, it makes a big difference to the structure of the product obtained. Applicants disagree with the Examiner's position at page 10, 1st and 2nd paragraph of the Examiner's Answer. Clearly, **a polymer that is post-crosslinked as disclosed in Modern Superabsorbent Polymer Technology (without being first admixed with a compound) is different from a polymer particle as obtained in the present**

invention in which a alkali metal silicate is distributed inside the polymer particle and which is then post-crosslinked.

Further according to the Examples of the present invention neutralization of the acid groups in the polymer particles occurs. In Example 1, a solution of soluble sodium silicate and sodium hydroxide is used wherein **74% of the acid groups of the hydrogel are neutralized.**

The result of the claimed invention are polymer particles **wherein the silicate is distributed throughout the particles** as shown in the Declaration of Dr. Manfred Essig dated May 6, 2005. The Declaration was filed at the USPTO on May 9, 2005.

Procter & Gamble refers to crystalline and amorphous silica that means particulate silica, only (page 5, second paragraph). **By mixing of different particulate materials it is impossible to get particles wherein the one material is distributed throughout the other material.** Thus, Procter & Gamble does not disclose a distribution of silica throughout the superabsorbent polymer particles (AGM).

Trinh et al discloses mixtures of superabsorbent particles and zeolite (column 25, lines 35-40). Kieselguhr can also be used (column 2, line 38). As shown in Kirk-Othmer "Encyclopedia of Chemical Technology" (IDS attached), "Kieselguhr" is a synonym for diatomite (page 109, first paragraph) and diatomite is a particulate material (page 108, first paragraph). **By wet mixing of different particulate materials it is impossible to get particles wherein the one material is distributed throughout the other material.** It is clear to a person of ordinary skill in the art that

in case of wet mixing, the particles would still be adjacent to each other and not one inside the other. That means that Trinh et al does not disclose a distribution of silica throughout the superabsorbent polymer particles.

Further, a person skilled in the art had no motivation to replace a porous silica or silicate with a high surface area (Procter & Gamble, page 5, 2nd and 3rd paragraphs) by an alkali metal silicate solution. Solvated alkali metal silicates cannot have any pores. The photographs attached to the Declaration does not show any porous silicate.

Modern Superabsorbent Polymer Technology has only been cited to show post-crosslinking and does not cure the defects of Procter & Gamble in view of Trinh et al.

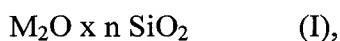
Even if Procter & Gamble, Trinh et al and Modern Superabsorbent Polymer Technology are combined, the present invention as claimed in Claim 1 cannot result.

Claim 10

Claim 10 relates to a process for preparing dried hydrogel particles, comprising:

polymerizing an olefinically unsaturated carboxylic acid or its salts in a polymerization reaction mixture, to obtain a solid gel containing a polymer;

admixing the polymerization reaction mixture before or during the polymerization or admixing said solid gel with an alkali metal silicate of the general formula I



wherein M is an alkali metal and n is from 0.5 to 4;

thereby obtaining particles of a gel;

postcrosslinking said particles of the gel; and

drying said particles of the gel at an elevated temperature, to obtain said dried hydrogel particles;

wherein said postcrosslinking is effected by a crosslinker which is a compound containing two or more groups that form covalent bonds with the carboxyl groups of said particles of the gel.

In the process of **Claim 10**, admixing of the polymerization reaction mixture is **before or during** the polymerization **or admixing the solid gel with an alkali metal silicate of the general formula I**, $M_2O \times n SiO_2$ (I), occurs.

The admixing of the polymerization mixture with alkali metal silicate has been discussed above for Claim 1 and applies here as well.

Regarding the phrase “solid gel”, Applicants wish to note the following. A solid gel does not mean that the same is dry. In fact, it is well known that a gel has a high water content. See also Modern Superabsorbent Polymer Technology at page 85 (bottom) referring to a “pasty material”, page 86 (top), referring to a “rubbery gel” and page 87 (bottom) “2.5 kg water must be removed for 1 kg of polymer” and the figure at page 91.

Thus, a person of ordinary skill in the art would know that the water content of a solid gel is high.

Further, the admixing the solid gel with an alkali metal silicate is followed by post-crosslinking. Procter & Gamble, Trinh et al lack a disclosure of post-crosslinking. While Modern Superabsorbent Polymer Technology generally discloses the use of post-cross-linking, there is no disclosure that a compound such as an alkali metal silicate is first mixed with a polymer to be post-crosslinked. However, it makes a big difference to the structure of the product obtained. Clearly, a polymer that is post-crosslinked as disclosed in Modern Superabsorbent Polymer Technology **(without being first admixed with a compound)** is different from a polymer particle as obtained in the present invention in which an alkali metal silicate is distributed inside the polymer particle and which is then post-crosslinked.

Even if Procter & Gamble, Trinh et al and Modern Superabsorbent Polymer Technology are combined, the present invention as claimed in Claim 10 cannot result.

Moreover, the Examples of the specification indicate that the hydrogels of the present invention have superior absorbency under load and gel layer permeability.

Example 1 has an absorbency under load of 20.9 g/g and a gel layer permeability of $3 \times 10^{-7} \text{ cm}^3\text{sec/g}$. After solvent and heat treatment for surface post-crosslinking, the absorbency under load is 24.7 g/g and the gel layer permeability is $60 \times 10^{-7} \text{ cm}^3\text{sec/g}$. See page 7, line 40 to page 8, line 11 of the specification.

Comparative Example 1 uses no sodium silicate and as a result the obtained product HAS NO GEL PERMEABILITY. After surface post crosslinking, the gel layer permeability is **three times lower** than Example 1 and only $20 \times 10^{-7} \text{ cm}^3\text{sec/g}$. See page 8, lines 15-25 of the specification.

Example 2 has, after surface post-crosslinking, an absorbency under load of 23.9 g/g and a gel layer permeability of $8 \times 10^{-7} \text{ cm}^3\text{sec/g}$. See page 9, lines 10-12 of the specification.

Comparative Example 2 uses no sodium silicate and has, after surface post crosslinking, a gel layer permeability which is **50% lower** than Example 2 and only 4 ($\times 10^{-7} \text{ cm}^3\text{sec/g}$). See page 9, line 22 of the specification.

Example 3 has an absorbency under load of 21.4 g/g and a gel layer permeability of $4 \times 10^{-7} \text{ cm}^3\text{sec/g}$ and centrifuge retention of 31.3 g/g. See page 9, lines 36-38 of the specification.

Comparative Example 3 uses no sodium silicate and has an absorbency under load of 20.9 g/g and a gel layer permeability which is **4 times lower** than Example 3 and only 1 ($\times 10^{-7} \text{ cm}^3\text{sec/g}$) and a centrifuge retention of 31.8 g/g. See page 10, lines 5-6 of the specification.

Moreover, **Examples 4-9** are superior to **Comparative Example 4** in absorbency under load and a gel layer permeability and centrifuge retention, as shown in Table 1 at page 11 of the specification.

Examples 10-15 are superior to **Comparative Example 4a** in absorbency under load and a gel layer permeability and centrifuge retention, as shown in Table 2 at pages 11 and 12 of the specification.

Examples 17-19 are superior to **Comparative Example 16** in absorbency under load and a gel layer permeability and centrifuge retention, as shown in Table 3 at pages 12 and 13 of the specification.

Examples 21-24 are superior to **Comparative Example 20** in gel layer permeability and centrifuge retention, as shown in Table 4 at pages 13 of the specification.

Example 25 has an absorbency under load of 21.6 g/g and a centrifuge retention of 30.0 g/g. After surface post-crosslinking, the absorbency under load is 24 g/g and the gel layer permeability is $62 \times 10^{-7} \text{ cm}^3 \text{ sec/g}$. See page 14, lines 21-38 of the specification.

Comparative Example 25 uses no sodium silicate and has an absorbency under load of 12 g/g and a centrifuge retention of 32.8 g/g. After surface post-crosslinking, the absorbency under load is 24 g/g and the gel layer permeability is $33 \times 10^{-7} \text{ cm}^3 \text{ sec/g}$ and the centrifuge retention is 28 g/g. See page 15, lines 4-14 of the specification.

Procter & Gamble, Trinh et al and Modern Superabsorbent Polymer Technology, alone or in combination do not suggest the superior results of the present invention obtained when using sodium silicate solution followed by post-crosslinking.

Claim 18

Claim 18 relates to dried hydrogel particles, prepared by

polymerizing an olefinically unsaturated carboxylic acid or its salt in a polymerization reaction mixture, to obtain a solid gel containing a polymer;

admixing said solid gel with an alkali metal silicate of the general formula

I

$$M_2O \times n SiO_2 \quad (I),$$

wherein M is an alkali metal and n is from 0.5 to 4;

thereby obtaining particles of a gel;

postcrosslinking said particles of the gel;

drying said particles of the gel at an elevated temperature, to obtain said dried hydrogel particles;

wherein said postcrosslinking is effected by a crosslinker which is a compound containing two or more groups that form covalent bonds with the carboxyl groups of said particles of the gel.

The admixing the solid gel with an alkali metal silicate is followed by post-crosslinking. Procter & Gamble, Trinh et al lack a disclosure of post-crosslinking. While Modern Superabsorbent Polymer Technology generally discloses the use of post-cross-linking, there is no disclosure that a compound such as a alkali metal silicate is first mixed with a polymer to be post-crosslinked. However, it makes a big difference to the structure of the product obtained. Clearly, a polymer that is post-crosslinked as disclosed in Modern Superabsorbent Polymer Technology (**without being first admixed with a compound**) is different from a polymer particle as obtained in the present invention in which a alkali metal silicate is distributed inside the polymer particle and which is then post-crosslinked.

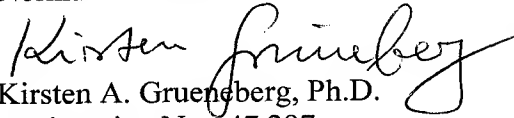
Even if Procter & Gamble, Trinh et al and Modern Superabsorbent Polymer Technology are combined, the present invention as claimed in Claim 18 cannot result.

Claims 4, 5 and 17 are dependent claims and are Not obvious over Procter & Gamble in view of Trinh et al for the reasons discussed in ground (A), above.

For the above reasons, it is respectfully requested that the rejections be
REVERSED.

Respectfully submitted,

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